Effect of carbon and tempering on dislocation density and hardness in lath martensitic steel with identical Ms temperature

Takuro Masumura[1], Keisuke Inami[2], Toshihiro Tsuchiyama[1], Shigenobu Nanba[3]

[1]*Department of Materials, Kyushu University, 744 Moto-oka, Nishi-ku, Fukuoka 819-0395, Japan*
[2]*Graduate school of Engineering, Kyushu University, 744 Moto-oka, Nishi-ku, Fukuoka 819-0395, Japan*
[3]*Materials Research laboratory, Kobe Steel, Ltd., 1-5-5, takatsukadai, Nishi-ku, Kobe 651-2271, Japan*

The dislocation density of lath α'-martensite, with the same Ms temperature and different C content, was evaluated by using the modified Williamson-Hall/Warren-Averbach (mWH/WA) method. Subsequently, the effect of carbon on the dislocation density of lath α'-martensite was clarified. Fe-18Ni, Fe-13.5Ni-0.13C, Fe-9Ni-0.26C, and Fe-0.55C alloys (mass%) were prepared. The Ni content of the alloys was adjusted to ensure that the Ms temperatures of the alloys were identical. The specimens were solution-treated at 1173 K for 1.8 ks, followed by water quenching to obtain the as-quenched α'-martensite structure. Then, some specimens were tempered at 573 K for 16.2 ks (tempered α'). Subsequently, the dislocation density was calculated using line profile analysis (mWH/WA method). In all the specimens, Ms temperatures were in the range of 550–600 K. In the as-quenched specimen, the dislocation density was overestimated due to tetragonality, and thus it is referred to as "apparent dislocation density". The overestimation of dislocation density increased with increasing carbon content and tetragonality. In tempered specimens, wherein the true dislocation density was obtained by removing the tetragonality, the dislocation density increased with carbon content even in the specimens with the same degree of auto-tempering. Additionaly, it was clarified that the dislocation density was almost saturated at 4–5 × 10^{15} /m^{-2} at a varbon content exceeding 0.15%.

Keywords: *martensite, dislocation density, solute carbon, tempering, line profile analysis*

1. Introduction

Lath α'-martensite is the main structure in high-strength steel, and it contains a large number of dislocations. The dislocation density of α'-martensite has been quantified via TEM [1)]. The results indicated that the dislocation density increases with increasing carbon content. However, in most specimens that were used for the experiment, the α'-martensite start (Ms) temperature of the specimens was varied with carbon content. This implies that the results would be also affected by recovery due to auto-tempering. Therefore, to accurately evaluate the effect of carbon, it is necessary to analyze the dislocation density by using specimens with same Ms temperature and different carbon content. Recently, modified Williamson-Hall/Warren-Averbach (mWH/WA) method, which can be used for X-ray line profile analysis, is often applied to calculate the dislocation density. The results of the previous study indicated that the tetragonality of the as-quenched carbon-bearing α'-martensite broadened the X-ray line profile, and thereby lead to an overestimation of the dislocation density [2)]. Furthermore, it was also observed that the dislocation density of as-quenched α'-martensite can be accurately evaluated by removing the tetragonality via low-temperature tempering at approximately 573 K wherein recovery hardly occurs. In this study, the dislocation density of lath α'-martensite, with the same Ms temperature and different carbon content, was evaluated by using the mWH/WA method. Subsequently, the effect of carbon on the dislocation density and hardness of lath α'-martensite was clarified.

2. Experiment

Commercially available 0.55 mass%C steel (0.55C steel) and 18 mass%Ni steel (18Ni steel), which have almost the same Ms temperature, were prepared. In addition, Fe-Ni-C alloys with various carbon contents and same Ms temperature (13Ni-0.13C, 9Ni-0.26C, and 5Ni-0.38C steels) were obtained by remelting the 0.55C steel and 18Ni steel in various ratios. The chemical compositions of the specimens are listed in **Table 1**. These specimens were solution-treated at 1173 K for 1.8 ks, followed by water quenching, and then immediately subzero-treated at 77 K for 1.8 ks in liquid nitrogen. Subsequently, some specimens were tempered at 373–873 K for different times in an oil bath or a salt bath. The *Ms* temperature was investigated by dilatometry using a Transmaster II instrument (Advance Riko, Inc., Japan) for φ3 × 10 mm specimens. The specimens were cooled from 1173 K at a cooling rate of 100 K/s.

Table 1 Chemical compositions of alloys used in this study(mass%).

	C	Si	Mn	Cr	Ni
18Ni	<0.001	<0.01	0.38	<0.01	17.88
13Ni-0.13C	0.13	0.36	0.45	0.17	13.37
9Ni-0.26C	0.26	0.70	0.53	0.35	8.86
5Ni-0.38C	0.38	0.93	0.61	0.53	4.51
0.55C	0.55	1.47	0.69	0.70	-

The microstructures of the specimens were examined by optical microscopy and electron backscatter diffraction (EBSD) analysis using a field emission scanning electron microscope (SIGMA 500, Zeiss, Germany) operated at an accelerating voltage of 20 kV with a step size of 300 nm. The solute carbon concentration in martensite was estimated by electrical resistivity measurements using the four-point method at 77 K [3,4)]. The square bar specimens for the electrical resistivity measurements had dimensions of 1 × 1 × 50 mm^3. After holding the specimens in liquid nitrogen for 60 s, the electrical resistivity was measured twice while changing the current direction, and the average

value was adopted.

The dislocation density was calculated using the mWH/WA method [5,6]. The plate specimens were cut to dimensions of 10 × 10 × 1 mm, polished with sand paper, and then electropolished in an acid mixture (H_3PO_4:CrO_3 = 2:1) by more than 50 μm in order to eliminate the effect of grinding-induced strained layers [7]. X-ray diffraction measurements with a Cu-Kα radiation source were carried out at 40 kV and 40 mA.

3. Result and discussion

Figure 1 shows the carbon content dependence of the M_s and M_f temperatures obtained by dilatometry test. Both the M_s and M_f temperatures show little dependence on the amount of carbon, and the maximum difference at the M_s temperature is only about 40 K. The M_f temperature show almost the same value, and it can be said that we have succeeded in producing carbon steels with same degrees of auto-tempering. **Figure 2** shows the crystallographic orientation maps of each specimen. The block size of α'-martensite becomes finer as the carbon content increases. This is because when the carbon content increases, the austenite matrix becomes harder, and the strain relaxation mechanism during α'-martensitic transformation is changed from the plastic accommodation of austenite to the self-accommodation, in which multiple α'-martensite variants are generated to relax the transformation strain [8]. Solid-solution strengthening by carbon and hardening due to a decrease in the M_s temperature have been thought to be the factors behind the hardening of the austenite matrix. **Figure 3** shows the carbon content dependence of the quenched α'-martensite hardness. As the carbon content increased, the hardness increased, and the value was the same as that reported for Fe-C alloys in the past.

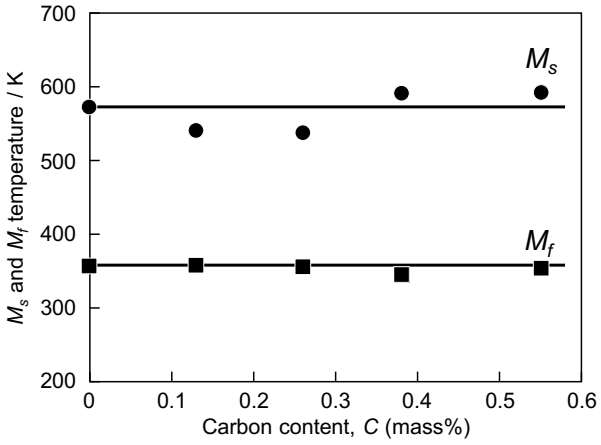

Fig. 1 Changes in M_s and M_f temperatures as a function of carbon content.

By tempering at 573 K for 16.2 ks, the overestimation of the dislocation density due to tetragonality and the carbon content dependence of the true dislocation density were investigated. **Figure 4** shows the apparent dislocation densities of the quenched specimens (black plots) and the dislocation densities of the 573 K–16.2 ks tempered specimens (white plots) derived from the line profile analysis. As the carbon content increased, the difference

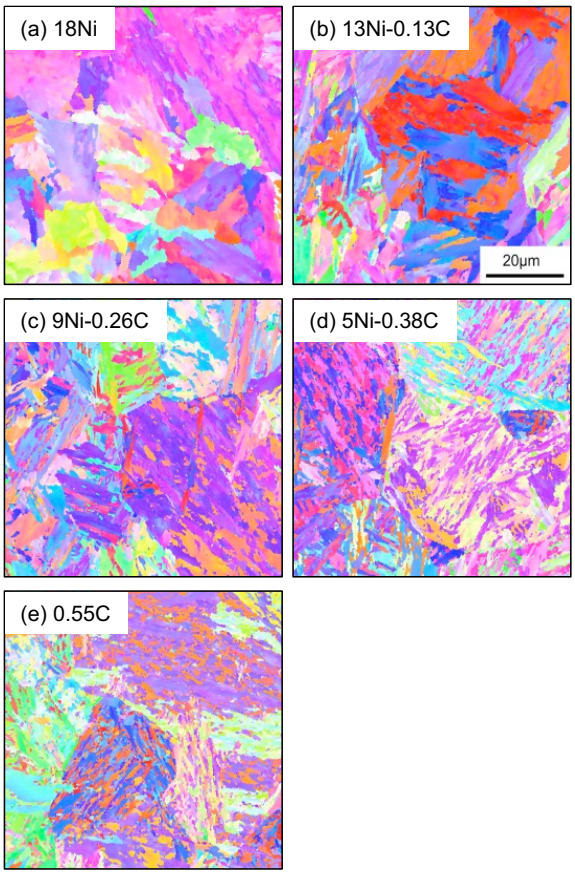

Fig. 2 Crystallographic orientation maps of Fe-Ni-C alloys.

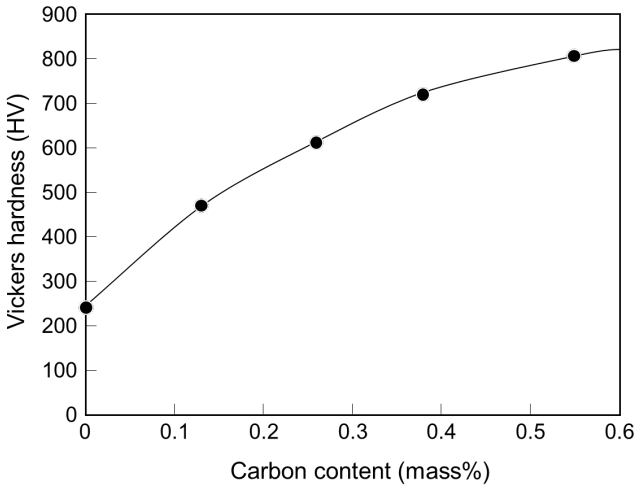

Fig. 3 Vickers hardness of as-quenched Fe-Ni-C alloys.

between the as-quenched and tempered specimens, that is, the overestimation amount due to the tetragonality increased. Focusing on the value of the tempered specimens, which indicates the true dislocation density, the dislocation density gradually increases as the carbon content increases, and becomes almost constant when the carbon content exceeds 0.3%. As for the carbon dependence of the dislocation density of martensite, Morito *et al.* [1] reported that the volume change in the α'-martensitic transformation increased with increasing carbon content, resulting in an increase in the number of dislocations required for lattice-invariant deformation. In

addition, it can also be considered that the dislocations introduced by lattice-invariant deformation are pinned by carbon atoms, leading to the introduction of many more dislocations to carry out the α'-martensitic transformation. In conclusion, even if the M_s temperatures were the same, the carbon content dependence of the dislocation density of as-quenched α'-martensite appeared clearly.

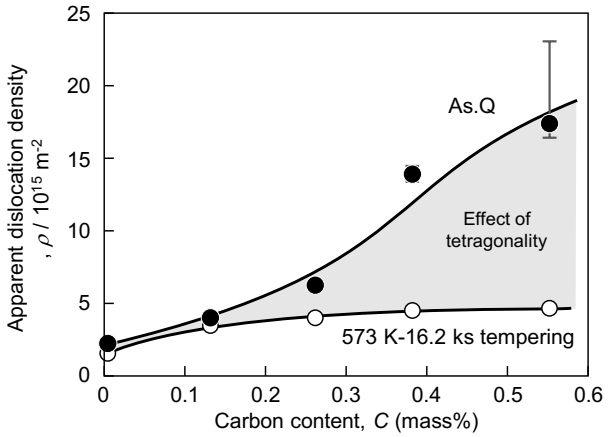

Fig. 4 Changes in apparent dislocation density and true dislocation density as a function of carbon content.

4. Conclusions

As the tetragonality increases with an increase in the carbon content, the overestimation of the dislocation density of quenched α'-martensite obtained by X-ray line profile analysis increases. As a result of measuring the true dislocation density corrected by tempering, it was clarified that the true dislocation density also increases with increasing carbon content even if the M_s temperatures are the same.

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